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AGRO INDUSTRIE RECHERCHES ET DÉVELOPPEMENTS

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

APPLICANT:	Bertho and Al
Serial N°:	10/008791
Filed:	01/02/2001
Title:	PROCESS FOR PREPARING A SOLUBILIZATION ADJUVANT FROM FUSEL OIL AND OSES
Examiner:	Travis C McIntosh
Art Unit:	1623

DECLARATION OF YVON LE HENAFF

I, Yvon Le Hénaff, hereby declare as follows:
I know the above-identified "791 application, and I am General Director of ARD (Agro Industrie Recherches et Développements), the assignee of the 791 application. I am presently, and I have been for the past twenty seven years, Agricultural and Food Industries Ingenior.
I have studied the Office action dated January 14, 2003 In the "529 application, and I am familiar with cited U.S. Patent 4,939,245 Rasche and U.S. Patent 6,087,403 to Bertho.
I have carried out the following tests.

A mixture of 600 g of Fusel oil (composed of water (10 %), ethanol (6 %), 2-propanol (0,2 %), 1-propanol (2,1 %), 2-methylpropanol (9,3 %), 1-butanol (0,3 %), 3-methylbutanol (45 %), 2-methylbutanol (20,3 %) and unidentified components (6,8 %)) and 170 g of D-xylose was formed at room temperature. The pressure on the mixture was reduced to 50 mbar and the temperature raised to 107°C during 1,5 hours. Distillation occurred and 50 g of alcohols blend is collected. 3,4 g of sulfuric acid is added to the mixture. The mixture became a dark heterogeneous syrup phase in 15 minutes. Separation of glycoside were impossible.

I further declare that all statements made herein of my own knowledge are true and that all statements made herein on Information and belief are believed to be true; and further that all these statements were made with the knowledge that wilful false statements and the like so made are punishable by fine or Imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such false statements may jeopardize the validity of the application or any patent issuing thereon.

Name LE HENAFF
Function General Director
Date August 18, 2003

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AGRO INDUSTRIE RECHERCHES ET DÉVELOPPEMENTS, S.A. au capital de 10 750 000 F. RCS Reims B 350 504 817 - Code APE 731 Z

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I have carried out the following tests.

This four tests consist In reacting sugar syrups with Fusel alcohols following the "Rasche" process (U.S. Patent 4,939,245) on the one hand and following the process described for preparing solubilization adjuvants from Fusel oils and saccharides (Appl. No. 10/008791) on the other hand.
In all the tests, the mixture of Fusel oil Is composed of water (10 %), ethanol (6 %), 2-propanol (0,2 %), 1-propanol (2,1 %), 2-methylpropanol (9,3 %), 1-butanol (0,3 %), 3-methylbutanol (45 %), 2-methylbutanol (20,3 %) and unidentified components (6,8 %)).

Test n° 1 : Synthesis of adjuvant from glucose syrup and Fusel oils following the "Rasche" process (dehydration of the carbohydrate source before addition of the acid catalyst)
A mixture of 150 g of D-glucose syrup, containing 37.5 g of water and 112.5 g of D-glucose and 400 g of Fusel oils was formed at room temperature. The pressure on the mixture was reduced to 50 mbar and the temperature raised to 107°C during 1,5 hours. Dehydration occurred and 75 g of hydrous alcohols blend is collected. The resulting mixture Is heterogeneous. Then, 2.25 g of sulfuric acid is added to the mixture. Two and half hours after catalyst addition, the reaction Is stopped and the mixture Is filtered. The 92.6 g of polar solids removed from the reaction are mainly composed of polyglucose and unreacted D-glucose.

The filtrate Is then neutralized and the unreacted Fusel oils are separated from the glycosides product by heating the mixture In a wiped film evaporator at 120 °C under reduced pressure (1 mbar). A low amount of Fusel polyglucoside product (21 g) is collected.

Test n° 2 : Synthesis of adjuvant from glucose syrup and Fusel oils following the process described In Appl. No. 10/008791 (dehydration during the acidic catalysed reaction)

150 g of D-glucose syrup, containing 37.5 g of water and 112.5 g of D-glucose are added dropwise over 1.5 hours to 400 g of Fusel oils containing 2.25 g of sulfuric acid, at 107°C. The water Is removed during the reaction by azeotropic distillation. After the addition of the sugar syrup, the homogeneous mixture Is gradually reduced to 900 mbar at the same temperature during 30 minutes. The homogeneous reaction mixture Is directly neutralized without further filtration and the unreacted Fusel oils are separated from the glycosides product by heating the mixture In a wiped film evaporator at 120 °C under reduced pressure (1 mbar). 121.7 g of Fusel polyglucosides are collected.

Test n°-3 : Synthesis of adjuvant from sugar blend syrup from wheat straw and Fusel oils following the "basket" process (dehydration of the carbohydrate source before addition of the acid catalyst)

A mixture of 150 g of sugar syrup derived from wheat straw and containing 37.5 g of water, 79 g of D-xylose, 13.6 g of L-arabinose, 8 g of D-glucose and 4.5 g of D-galactose and D-mannose and 400 g of Fusel oils was formed at room temperature. The pressure on the mixture was reduced to 50 mbar and the temperature raised to 107°C during 1,5 hours. Dehydration occurred and 71 g of hydrous alcohols blend is collected. The resulting mixture Is heterogeneous. Then, 2.25 g of sulfuric acid is added to the mixture. Two and half hours after catalyst addition the reaction Is stopped and the mixture Is filtered. The 82 g of polar solids removed from the reaction are mainly composed of unreacted sugar.

The filtrate Is then neutralized and the unreacted Fusel oils are separated from the glycosides product by heating the mixture In a wiped film evaporator at 120 °C under reduced pressure (1 mbar). A low amount of Fusel polyglycosides (30 g) is collected.

Test n° 4 : Synthesis of adjuvant from sugar blend syrup from wheat straw and Fusel oils following the process described In Appl. No. 10/008791 (dehydration during the acidic catalysed reaction)

150 g of sugar syrup derived from wheat straw and containing 37.5 g of water, 79 g of D-xylose, 13.6 g of L-arabinose, 8 g of D-glucose and 4.5 g of D-galactose and D-mannose are added dropwise over 1.5 hours to 400 g of Fusel oils containing 2.25 g of sulfuric acid, at 107°C. The water Is removed during the reaction by azeotropic

distillation. After the addition of the sugar syrup, the homogeneous mixture Is gradually reduced to 900 mbar at the same temperature during 30 minutes. The homogeneous reaction mixture Is directly neutralized without further filtration and the unreacted Fusel oils are separated from the glycosides product by heating the mixture In a wiped film evaporator at 120 °C under reduced pressure (1 mbar). 125 g of Fusel glycosides are collected.

Conclusion :

The following figure show the efficiency of the process described In the Appl. No. 10/008791 In regard with the "Rasche" process.

The "Rasche" process result In low yield of Fusel glycoside, high amount of sugar waste and long reaction time (4 hours).

Tests	1	2	3	4
Process	"Rasche"	Appl. No. 10/008791	"Rasche"	Appl. No. 10/008791
Sugar source at 75 % dry matter	150g of D-glucose syrup	150g of D-glucose syrup	150g of Wheat straw sugar syrup	150g of Wheat straw sugar syrup
Filtration	Yes	No	Yes	No
Residual waste after filtration	32.6 g	0 g	52 g	0 g
Overall time of the reaction	4 hours	2 hours	4 hours	2 hours
Fusel glycosides products	21 g	121.7 g	30 g	125 g

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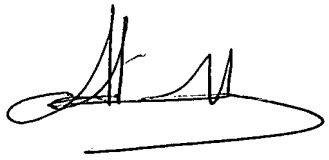
Name LE HENAFF
Function Managing Director
Date September 4, 2003



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I have carried out the following tests.

The tests have been carried out following a procedure described in the publication of S. Ikeda and Y. Maruyama, *Journal of Colloid and Interface Science*, 166,1-5,1994.

This method consist in making a dye (Sudan Red 7B) partially soluble in an aqueous solution of surfactant micelles and determining the amount of soluble dye by spectroscopic measurements after isolation of the dye and destruction of the micelles.

Procedure description :
An excess of dye is added to a solution of surfactant. The solution is stirred vigorously at 25°C for 30 minutes. After this equilibrium period, the excess of dye is filtered out on cellulose acetate membrane (0.2 µm, GELMAN) and the filtrate is diluted in 50 ml of ethanol.
Ethanol is a suitable solvent for micelles destruction and liberation of soluble dye. Then, 535 nm absorbance measurements (with a Hitachi U2000 spectrometer, in a 1 cm plastic tank) of the ethanol solutions give the amount of dye soluble.

Results :
The following graphic shows the synergistic effect between Fusel glycoside of the invention and alkyl glycoside detergents (octyl-decyl wheat bran glycoside following the french patent n° FR 2723858) in the solubilization of Sudan Red dye.

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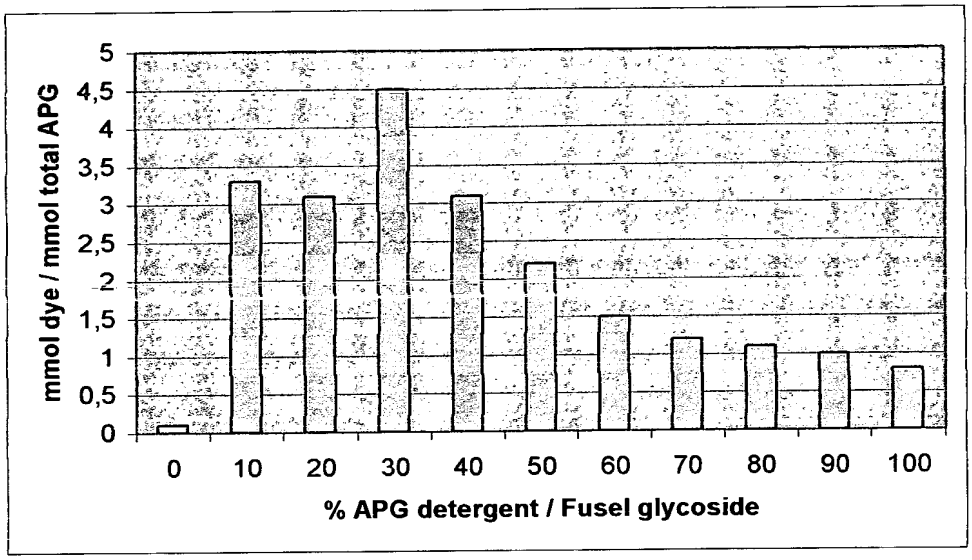
Synergistic effect on solubilization of Sudan Red with Fusel glycosides solution of APG detergent.



On the precedent graphic we show that a solution of pure Fusel glycosides or of pure alkyl polyglycoside detergents only permit less than 1 mmol of dye to get soluble. Whereas mixing alkyl polyglycoside detergents with Fusel glycosides allows the solubilization of high amounts of dye.

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